9T Horizontal Field Magnet Operation Manual

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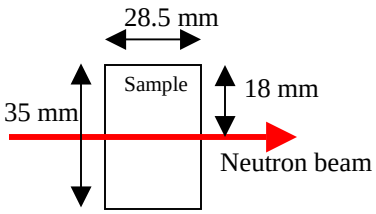
I. Prepping

1. Pump on outer vacuum space to a pressure of 10^{-5} torr. (Not necessary if the system has been used recently.)
2. Pump out the sample space using a turbo pump ($< 3x10^{-4}$) unless you are 100% certain there is either a good vacuum or only He inside.
3. Using a rough pump, pump out the annulus with the needle valve closed and purge with He gas to prevent condensation from forming during the cooling process. Repeat the process twice more. The last time sealing the He gas in the annulus. Then do the same thing to the liquid helium reservoir, the last time sealing the He gas in it.
4. Leaving the needle valve and annulus exhaust valve closed and opening the He exhaust ball valve and the He fill port fill the He reservoir with LN_2 and let cool to 77K. The magnet temperature sensor will have a resistance of ~7.6 Ω at this point. After one hour fill the liquid nitrogen reservoir as well. When the liquid nitrogen reservoir is full stop filling both reservoirs and check the level in the liquid helium reservoir using a bakelite stick. If there is greater than five inches of liquid nitrogen close the two open valves and let the system sit over night. If not continue filling another 15 minutes then check again, continuing until there is greater than 5 inches of liquid nitrogen present.
5. When the magnet has cooled to nitrogen temperature, the remaining nitrogen needs to be blown out. The best way to do this is to blow warm He gas, at about 5 psi, in through the He exhaust port. There is a piece of stainless steel tubing with a Teflon tip that is attached to the magnet cart, attach a rubber hose to the end and place in the He fill port. The nitrogen will spray out here, so you may want to catch it in a trashcan or small dewar. Remove the large brass relief valve from the He exhaust and replace with a blank-off flange to build up enough pressure.
6. Continue to blow the warm helium through until the magnet temperature has risen to ~90K (9-10 Ω) to be sure all the nitrogen has been removed. You may use the magnet heater to boil off the last bit of liquid nitrogen. The current through the 50 Ω heater should not exceed 1 Amp.
7. Replace the large brass relief valve and then, as in step 3, pump purge the He reservoir with He gas three times, the last time sealing the He gas in it.
8. Now you can fill the helium reservoir with liquid helium. There should be a brass fitting in the small parts box that needs to be put on the end of the transfer line that goes into the cryostat. (This is because our transfer tubes are smaller than the fill port and we don't want He spraying out of that port.) Make sure the He exhaust ball valve is open. Shortly after starting the fill open the needle valve, begin pumping on the annulus with a rough pump and slowly open the annulus valve. This will pull a steady flow of He gas through the needle valve to prevent it from blocking. Be careful not to overwhelm the pump, obvious by the smell of oil and or loud noises, by closing down the needle valve almost all the way.
9. You can monitor the progress with the He level sensor. The meter measures in millimeters and the reservoir is full when the sensor reads 240mm (anything greater than that is an erroneous reading that is equal to 240mm).
10. Refill LHe when the level gets below 40mm and top-off the liquid nitrogen every time you fill helium.

II. Procedure for Loading Sample in 9-T Horizontal Field Superconducting Magnet

1. Make sure the gate valve is closed.
2. Mount sample onto sample probe. Mark sample height for clamp. If the sample is a single crystal, also mark orientation. Grease the whole length of the sample stick to prevent leaks from forming around the o-rings in the green fitting.



Distance to beam from top of green flange on sample stick = 1363 mm
Distance to beam from bottom of sample stick = 18 mm
Max diameter = 28.5 mm
Max height = 35 mm
Mounting Surface: 8- M3 tapped holes on 24.0mm diameter and 5/16"-18 tapped hole in center

3. Put the sample stick in the airlock, which should already be on the magnet, and tighten the clamps. Adjust clamp on sample stick so that sample is held within airlock.
4. Connect the rotary or turbo vacuum pump to the airlock evacuation valve. Pump out airlock to 1 x 10^{-4} mbar and then close airlock evacuation valve.
5. Open the gate valve.
6. Put the sample stick in slowly and adjust the height with the height adjustment clamp.

III. Procedure for Unloading the Sample

1. Loosen the height adjustment clamp.
2. Slowly pull the stick all the way up. If the sample well has been cold, you may want to stop and let it warm up several times along the way. The stick may freeze the O-rings and get stuck. If this happens, use a heat gun to gently warm the stick. Do not force the stick through the O-rings, or they may crack and cause leaks.
3. Move clamp on stick down to load lock and tighten.
4. Close the gate valve.
5. Connect the helium gas to the airlock evacuation valve and fill the airlock with helium gas.
6. Allow the sample to warm. There should be no condensation on the airlock when the sample is ready to be removed.
7. Loosen the upper airlock clamp and remove the stick.
8. Cap the airlock with a blank flange.
9. Pump out airlock to 1 x 10^{-4} mbar and then close airlock evacuation valve.

IV. Procedure for Adding Exchange Gas

It may be necessary to add some helium exchange gas to the sample well. This is done once when the magnet is first cooled down. After that, it may be necessary to add helium due to the loss of exchange gas after multiple sample changes. If the sample temperature and the control temperature are not in reasonable agreement, then adding helium exchange gas should be the solution.

The sapphire windows in the sample well are fragile, and easily dislodged by over-pressurizing the sample well! Exchange gas is always introduced in small, controlled amounts.

1. Adjust the regulator on the helium supply to ~2 psi outlet pressure.

2. With helium flowing to purge the line, attach the helium hose to the closed airlock evacuation valve.

3. Pinch off the helium hose tightly approximately 6 inches from the airlock evacuation valve. This trapped volume is the amount that will be introduced to the sample well.

4. Open the airlock evacuation valve to suck in the trapped volume of helium and then close it again. There may be a momentary rush of cold helium gas from the helium reservoir when this action is performed.

5. If the sample well is warm and evacuated, add enough exchange gas to bring the pressure up to 10in Hg, which is less than 1 atm, or 0 psi on the pressure gauge.

6. Repeat steps 3 and 4 if the sample and control temperatures do not come into reasonable agreement within 5-10 minutes.

7. As the sample well cools, the pressure will drop, due to the ideal gas laws, and the gauge will read 30in Hg (which is the bottom of the gauge), but there is still gas in there. When you add gas, you may not see the pressure rise on this gauge, so be careful not too add too much.

8. If you add exchange gas while the sample well is cold, keep an eye on the pressure as it warms up. Make sure the pressure does not rise above 1 atm. If it is getting close, pump on the sample well until it comes back down.

V. Operation of the Cryogenic, Ltd. Magnet Power Supply

1. Make sure leads are securely connected and the persistent switch heater is plugged in before turning on power supply.
2. Turn on power supply. The ZERO button will be the only button lit. The screen should read "Controller Version 4.30". If there is a fault message displayed, consult the user's manual found on the magnet cart. To enter a set point, press SELECT until you see "MID Set Point = x.xxxx Amps". Enter a destination value using the keypad and press enter. Do not change the MAX setting (which should read 119A or 9.0T) or the RAMP RATE (0.09 A/s).
3. Turn on the persistent switch heater by pressing the HEATER button. This light will come on when the heater is on. Wait 30 seconds before proceeding to the next step.
4. Start ramping to the set point by pressing the MID button. You can halt the run at any time using the PAUSE button.
5. When the magnet reaches the desired current, turn off the heater to put the system in persistent mode. The power supply will remember the current at which the heater was turned off and display it on the screen.
6. Wait 30 seconds and then press the ZERO button to ramp the leads to 0 Amps. Once the leads are ramped down, the power supply will read 0.00 Amps, but the magnet is still at field, in persistent mode.
7. To change fields, or turn off the magnet, ramp the leads back up to the current where the heater was switched off. You can do this by pressing the MID button, which is still set to the previous current.
8. Press PAUSE to halt the run. This will keep the magnet from going to the new set point before you are ready. Enter the new field using the SELECT button to change the MID setting, as before.
9. Turn the heater back on and wait 30 seconds. Press PAUSE again to begin ramping to the new set point. If you want to turn off the magnet, simply hit the ZERO button, then PAUSE to start the ramp down.
10. When you are finished, make sure that the magnet has been ramped to zero field, not just the leads.

The screen will look something like this most of the time:

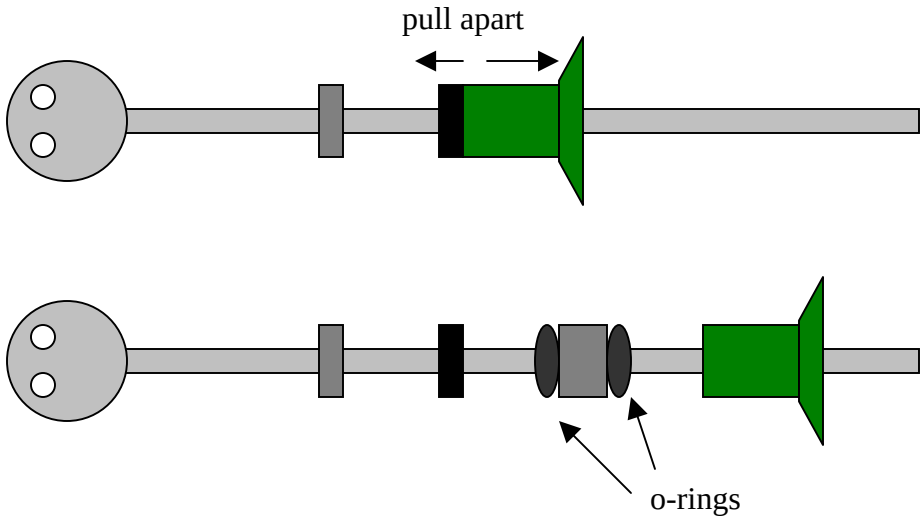
1.24 Amps		SMS120C-H	0.00 Volts
35.0 Amps	119 Amps	4.0 Volts	0.09 A/s

The numbers correspond to these parameters:

Current			Voltage
MID Set Point	MAX Set Point	Voltage Limit	Ramp Rate

VI. If a leak develops in the sample well:

1. Remove the sample stick and cover sample well with blank flange.
2. Remove three bolts on the top of the green fitting and pull apart.
3. Inside are two o-rings, separated by a spacer. Grease the o-rings and reassemble.
4. Make sure the length of the sample stick is greased before inserting it into the sample well.
5. Follow sample loading procedure as usual.



Note: Symptoms of a sample well leak include: stuck sample stick, inefficient cooling in sample well, and increased pressure in sample well.

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VII. Temperature Control:

Temperature control is done using a Lakeshore 340 controller that has two sensor inputs and two heater outputs. There are two sensors that are used during operation of the system both of which are Cernox type sensors. The first is located on the VTI heat exchanger is used for control. The second is located near the bottom of the sample stick and is used for a more accurate reading of actual sample temperature. Similarly there are two heaters on the system as well. One is located on the VTI heat exchanger and is a 50 ohm and 1 amp max heater. The other is located near the bottom of the sample stick and is a 100 ohm and 100mA max heater. The VTI heater is used for normal control of the temperature over the entire temperature range of the system. The sample stick heater, when necessary, is used for fine control at lower temperatures

1. Turn on the controller using the button on the right side of the back of the unit. Verify which sensor, VTI or Sample, is connected to which input, A or B, by reading the labels on the cables and the back of the controller.
2. Plug in the VTI Heater cable single banana plugs into the High and Low plugs on the back of the controller
3. Set the control channel to the VTI sensor by pressing the Control Channel button, selecting the appropriate channel and then pressing Enter.
4. Set the desired temperature by pressing the Setpoint button, then entering the temperature using the number pad and finally pressing Enter.
5. Set the correct heater range by pressing the Heater Range button, selecting a range using the Up or Down Arrow buttons and then pressing Enter. The temperature range you are working in should determine the heater range. The maximum heater range is needed to reach room temperature, but lower temperatures may only require, and may have better control, using a lower range.
6. You know the heater is on by seeing a non-zero percentage of the heater power on the display under the heater range. Keep in mind that is if the temperature of the sensor is above the set point the heater will not turn on until necessary.
7. If the temperature is not dropping to your set point it may be necessary to open the cold valve on the top of the cryostat to increase the cooling power. The valve can be opened wide for a quick cool down over a large range, but should always be closed as much as possible once the set point is reached to conserve helium.
8. There will almost always be some difference between the sensors even after staying at one temperature for a long time. The best thing to do is to use the VTI for control and the sample as the actual temperature. This is because the VTI sensor is placed near the heater and controls the temperature of the bottom of the sample well that shields the sample. If a specific sample temperature is needed simply offset the set point on the VTI to compensate.
9. The sample stick heater can also be used if none of the above solutions works. Please contact Evan Fitzgerald (x6657, evan@nist.govT) before attempting this.